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L2: Entry 16 of 51

File: USPT

Mar 6, 2001

DOCUMENT-IDENTIFIER: US 6197201 B1

**** See image for Certificate of Correction ****

TITLE: Process for removal and stabilization of arsenic and selenium from aqueous streams and slurries

Detailed Description Text (2):

As used herein, "removing an ion from a solution or slurry" refers to reducing the concentration of such ion, preferably arsenite, arsenate, selenite or selenate ion, preferably to a concentration determined by the goals of the process. "Aqueous solution" refers to a solution in which water is the dissolving medium or solvent. "Slurry" refers to suspended solid particles including different ores and rocks in a liquid which contains water. A "precipitating composition" refers to any of the agents described herein that cause precipitation of the ions of interest. A "solid" material refers to the resultant material formed from the contacting of the precipitating composition with the aqueous solution or slurry. Solid materials formed include amorphous materials and crystalline materials or mixtures. The pH of the aqueous solution or slurry is adjusted by any means known by one skilled in the art, including addition of calcium hydroxide or sodium hydroxide to raise the pH or acid to lower the pH. A "concentration effective for removing at least one ion" refers to the concentration of precipitating composition required to remove a measurable amount of the selected ion. A "flocculating agent" is a chemical that induces the formation of particles suitable for precipitation. One example of a flocculating agent is BuFloc (Buckman Laboratory, Memphis, Tenn.). Adjusting the oxidation potential of the aqueous solution or slurry can be achieved by any means known to those skilled in the art, including the addition of bleach (sodium hypochlorite) to the solution. "Stabilizing an ion" refers to the process of making the ion resistant to a number of processes including leaching, degradation and other decomposition processes and does not necessarily include precipitating the ion. "Lanthanum chloride" refers to both pure and impure lanthanum chloride. Impure lanthanum chloride can contain various elements of the lanthanide series in addition to lanthanum. The lanthanide series of elements includes the elements lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium and lutetium.

Detailed Description Text (57):Stabilization of Arsenic in Solids of Minahasa Tailings Slurry in Sea WaterDetailed Description Text (58):

The tailings from Minahasa Indonesia gold mine operations is submarine discharged into the ocean. Therefore it is necessary to determine the stability of arsenic to sea water after treatment of the tailings slurry. An experiment was conducted using the following conditions:

Detailed Description Text (60):Initial Sea Water pH=7.72Detailed Description Text (61):Initial As concentration in Sea Water=<0.05 ppb.Detailed Description Text (62):

To a 20 gram sample of stabilized slurry (dry), 180 ml sea water was added. The slurry was stirred gently at 30 to 35 rpm. After 1 hour, 4 hours and 8 hours, the pH of the slurry was measured. At each time, 30 ml of slurry was filtered with 0.45 micron filter paper and the arsenic concentration in the filtrate was analyzed.

Detailed Description Text (63):

Experimental results of arsenic release as a function of time are given in Table 12. It can be seen that the arsenic concentration of as-received dried tailings which leached out in both sea and tap water were around 9 ppm after 8 hours. This observation reinforces the notion that arsenic is continuously released from the solid phase. Table 12 clearly shows that the slurry stabilized with combination of lanthanum chloride and ferric sulfate is strongly resistant to releasing arsenic upon exposure to seawater. In this case the arsenic concentration was about 3.98 ppm after 8 hours.

Detailed Description Paragraph Table (13):

TABLE 12 Stability of Arsenic in Solids of Minahasa Tailings Slurry 1 hour 4 hours 8 hours Dosage As As As Treatment lb/ton pH ppm pH ppm pH ppm Untreated Solids 8.5 6.14 8.3 8.43 8.2 9.02 from Slurry in tap water Untreated Solids 8.1 5.35 8.1 7.45 8.1 8.9 from Slurry in sea water Fe.sub.2 (SO.sub.4).sub.3 2.0 8.0 4.92 8.1 5.5 8.1 6.37 LaCl.sub.3 0.5 8.1 5.12 8.1 5.74 8.1 5.96 Fe.sub.2 (SO.sub.4).sub.3 + LaCl.sub.3 2.0 + 0.5 8.0 3.63 8.1 3.91 8.0 3.98

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L2: Entry 21 of 51

File: USPT

May 6, 1997

DOCUMENT-IDENTIFIER: US 5626825 A

TITLE: Process for the purification of magnesium hydroxide

Brief Summary Text (2):

Magnesium hydroxide is mainly obtained by cave extraction, by sea brine extraction, by reaction with NaOH or Ca(OH)₂, or by sea water extraction, making the magnesium hydroxide therein contained precipitate with a strong base and then extracting by known techniques (see the booklet of Sardamag Spa, January 1994-- "Production of sea water magnesite for refractory purposes", Moscuza et al.; Villavecchia Eigenmann: vol. 4, pag. 2006).

Brief Summary Text (3):

The natural magnesium hydroxide extracted from caves shows a relatively high purity of 96-98%; at present the low extracting cost, the great availability and the high purity make this product preferable in respect with the product extracted by sea water, for some peculiar applications or as starting material for further purification; however the qualitative and crystalline unevennesses make this source limiting as to pharmaceuticals and as flame retarder, in the industry of plastics and cables and also in the industry of cosmetics and detergents.

Brief Summary Text (5):

The magnesium hydroxide synthesised from sea water is obtained by precipitation with a base, for instance calcium or sodium hydroxide, effecting consecutive washings with inorganic acids of low price, such as for instance HCl, H₂SO₄, and then solubilizing again the hydroxide and recrystallizing it with the help of great quantities of washing water, thereby obtaining improvements of the titer.

Brief Summary Text (9):

The anions normally contained in sea water, which are not eliminated during the purification process, obviously reduce the titer of the final product.

Brief Summary Text (10):

Further, magnesium hydroxide easily reacts with the carbon dioxide present either in the water or in the air forming magnesium carbonate (and eventually calcium carbonate if calcium hydroxide is used for the precipitation) and thereby further lowering the purity thereof: the titer of magnesium obtained is in fact hardly higher than 93/95% (expressed as MgO).

Brief Summary Text (12):

A thermic process, where the hydroxide is calcined and transformed into the oxide and then rehydrated to be retransformed into the hydroxide, is used to further purify magnesium hydroxide from sea water.

Brief Summary Text (19):

The starting product can be magnesium hydroxide extracted from sea brine or from sea water just prepared or even packed and being some months old.

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L2: Entry 22 of 51

File: USPT

Mar 26, 1996

DOCUMENT-IDENTIFIER: US 5501798 A

TITLE: Microfiltration enhanced reverse osmosis for water treatment

Detailed Description Text (4):

Thus, for example, by reference to FIG. 1, there is provided a schematic illustrating the process of the invention utilizing a high recovery RO membrane followed by a small microfiltration membrane. In FIG. 1, there is provided a container 2 into which waste water stream 4 is introduced. If stream 4 contains suspended solids, a prefiltration device such as an MF membrane, sand-bed filtration with or without flocculant addition, dead-end cartridge filtration or a combination of these may be used to remove the suspended solids that may foul the RO membrane. In this process, an antiscalant 6 may be introduced to container 2, if desired. Liquid having soluble and sparingly soluble constituents is withdrawn from container 2 along line or conduit 8 and pumped along pipe 12 by high pressure pump 10 to reverse osmosis unit or chamber 14. The liquid is introduced to high pressure side 20 of RO membrane chamber 14. In chamber 14, the RO membrane rejects the soluble and sparingly soluble constituents such as soluble salt to provide purified liquid or permeate on low pressure side 22 of the RO membrane. The permeate is removed along line 16. By rejecting the soluble and sparingly soluble constituents, the RO membrane provides a retentate that is removed along line 18. The retentate is removed from the high pressure side of the RO membrane at a rate sufficient to avoid precipitation of the sparingly soluble constituent on the membrane with its attendant problems. That is, the rate of removal from high pressure 20 is important to avoid precipitation on the membrane and to avoid interference with its ability to operate at high efficiency. The retentate can contain high levels of the sparingly soluble constituent or even be supersaturated as long as its removal is effected at a rate that avoids precipitation. After removal of the retentate along line 18, a precipitating agent such as sodium hydroxide, magnesium hydroxide, ammonia, sodium carbonate, calcium hydroxide and like chemicals that will reduce the solubility of the sparingly soluble salts, is added that effects precipitation of the soluble constituent. The precipitating agent may be added to high pressure side 28 of chamber 24 as long as precipitation is effected.

Detailed Description Text (20):

A system for purifying wastewater was set up substantially as shown in FIG. 1. For purposes of testing the system, a synthetic feed water was formulated using city water by the addition of calcium carbonate, calcium sulphate and silica. The feed water contained total dissolved solids (TDS) 1200 mg/l, 870 mg/l SO₄, 230 mg/l Ca, 90 mg/l HCO₃, 45 mg/l SiO₂. A polymeric antiscalant compound (polyacrylic acid-2enotreat 150) was added at a concentration of 20 mg/L to the influent water. The process was operated for 12 days. The RO membrane was a 2.5-inch high pressure seawater polymeric spiral-wound membrane module available from Film Tec Corporation, Minneapolis, Minn., under the designation FT-30 SW 2540. The MF membrane was a Zenon hollow fiber polymeric microfiltration membrane available from Zenon Environmental, Inc., Burlington, Ontario, Canada under the designation ZW-10. The system was operated in a semi-continuous mode wherein spike city water was fed continuously to the RO membrane as in FIG. 1, and the RO permeate was continuously discharged to drain while returning the RO concentrate to container 2 via MF filtration unit 24. In this way, the concentration of the soluble and sparingly soluble materials increased as the water recovery increased. At 85% RO

recovery, concentrations of the retentate in the RO tank at a pH of 8.7 were as follows: TDS was 5700 mg/l, SO.sub.4 was 3300 mg/l, Ca was 1200 mg/l, HCO.sub.3 was 330 mg/l and SiO.sub.2 was 174 mg/l. When this concentrate was transferred to a precipitation tank and the pH adjusted to 9.9 by adding sodium hydroxide, the TDS was 4900 mg/l, SO.sub.4 was 3000 mg/l, Ca was 770 mg/l, HCO.sub.3 was 72 mg/l and SiO.sub.2 was 38 mg/l. The RO permeate had a TDS of only 6 mg/l. Thus, it will be seen that the concentrations of sparingly soluble materials (especially HCO.sub.3 and SiO.sub.2) were significantly reduced. The total suspended solids (TSS) was increased from 12 ppm to 900 ppm indicating precipitation of said materials external to the RO membrane. The MF membrane flux was 50-55 gallons/sq. ft./day (gfd) at 15.degree.-20.degree. C. and at a slight vacuum of -6 psig and the RO flux was steady at 12-15 gallons/sq. ft./day (gfd) at 20.degree. C. and 400 to 500 psig. These steady permeate flux data show that both the MF and RO membranes did not foul or scale at 85% to 90% recovery, in spite of the presence of high concentrations of sparingly soluble materials in the incoming water. As the overall recovery reached approximately 91%, the RO membrane flux started to decrease but only slowly, reflecting the onset of solids precipitation which is believed to be due to the high silica concentration in the RO membrane tank. Thus, it will be seen that the system can be operated at very high percent recovery without any significant decrease in flux.

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L2: Entry 30 of 51

File: USPT

Jul 31, 1984

DOCUMENT-IDENTIFIER: US 4462713 A

TITLE: Method for mining and reclaiming land

Brief Summary Text (22):

The average 4% salt level in soil indicates approximately 100,000 lbs. of salt are present per foot acre in the Highwood Bench area. It is been found that the most common salts are sodium and magnesium sulfates and that magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) is the predominant salt in the saline seep soils. Magnesium sulfate has a fairly high solubility of 262 grams per liter and as a result, it is easily leached from the soil and is concentrated in the water table. Magnesium sulfate is highly toxic to plant life and in view of a relatively high concentration of about 3,000 to 4,000 mg. per liter, as opposed to sea water which contain approximately 1,300 mg. per liter, is practical to mine commercially from the saline seep water.

Brief Summary Text (23):

The present combined method of mining and reclaiming land of the invention contemplates the collection of the saline seep and its treatment to first remove available calcium as calcium carbonate to prevent contamination of the magnesium. One such method of pretreating the saline seep to remove available calcium as calcium carbonate would be to treat the raw saline water with sodium carbonate and sodium hydroxide to remove calcium carbonate. Thereafter, magnesium can be removed as magnesium hydroxide by treating the solution with sodium hydroxide to precipitate and remove the magnesium hydroxide from the filtrate for further treatment. Calcium hydroxide or lime is then added to the resulting filtrate to remove the calcium sulfate as gypsum. The magnesium hydroxide and calcium sulfate both have commercial uses which make the mining of the saline seep water economically justifiable.

Brief Summary Text (30):

The combined mining and land reclamation process of the invention would in its preferred mode utilize a plastic lined holding pond or tank to prevent the penetration of the brackish collected drainage waters from penetrating into the ground. The water in these holding ponds or tanks could then be periodically diverted into further tanks for treatment with sodium carbonate Na_2CO_3 and sodium hydroxide NaOH to react with the calcium in the brine and form calcium carbonate which could be precipitated or filtered out of the solution from the bottom of the pond or holding tank. The filtered solution or mother liquid could then be transferred to another holding tank or pond where additional sodium hydroxide would be added to precipitate out magnesium hydroxide which would be collected and thereafter processed to produce magnesium metal. The mother liquid or filtered solution could then be transferred to a further tank or holding pond and calcium hydroxide would be added to precipitate further calcium as a calcium sulfate. The drained off mother liquid or filtrate could then be transferred to a further holding tank or pond and treated with sodium carbonate to remove excess calcium as a calcium carbonate precipitate before the water is further purified or returned to the recharge or discharge area depending upon the soil salinity in the respective areas.

Detailed Description Text (12):

Referring now to FIGS. 2 and 3, the combined method of mining and reclaiming land is illustrated in which a saline seep is controlled by the utilization of drainage pipe 30 to collect terranean and subterranean waters from a saline seep 24. The subterranean waters from water table 14 as represented by arrow 26 (FIG. 1) is collected by drain pipes 30 along with terranean water supplied from irrigation source arrows 28. Raw saline seep water that has been collected from areas in Montana as will be set forth hereinafter in greater detail in Examples contains more dissolved solids than sea water. Sea water typically contains about 36,000 mg. per liter of total dissolved solids whereas the total dissolved solids of brackish waters collected in sample areas have been as high as 49,700 mg. per liter.

Detailed Description Text (13):

The collected raw saline seep water as represented by block 40 is then treated with sodium carbonate and sodium hydroxide to remove calcium carbonate as represented by block 42 to prevent contamination of the magnesium hydroxide salts. As has been discussed, raw saline seep water can be collected in a first pond or tank and thereafter fed to a processing tank for treatment with sodium carbonate, sodium hydroxide to precipitate out the calcium carbonate which may be filtered or settled out from the remaining liquid which can then be transferred to a second tank for the removal of magnesium by the precipitation of magnesium sulfate as magnesium hydroxide by the treatment of the filtrate represented by block 46 with sodium hydroxide to precipitate out the magnesium hydroxide represented by block 48 which is subsequently treated and reduced to magnesium metal in a process that will be described hereinafter in greater detail.

Detailed Description Text (16):

In accordance with the present combined method of mining and reclaiming land it has been found that magnesium can be removed from brackish water with only minimal additions of sodium hydroxide as will be discussed in the examples hereinafter in greater detail. The reverse osmosis unit is effectively utilized after pretreatment and may be a unit manufactured by Osmonics, Inc. of Hopkins, Minn. or unit manufactured by Allied Water Corp. of San Francisco, Calif. Alternatively ion exchange, which will be discussed hereinafter in greater detail or electrodialysis may be advantageously employed after pretreatment to purify the water to potable condition. Electrodialysis units are available on the market and can be purchased from Ionics, Inc. of Watertown, Mass.

Detailed Description Text (17):

Referring now to FIG. 3, a process for the recovery of magnesium from the magnesium hydroxide precipitate of FIG. 2 is illustrated. The magnesium hydroxide as represented by block 48 is placed into a settling tank 80 and is subsequently filtered through a filter 82 to which filtrate hydrochloric acid represented by block 84 is added to form a magnesium chloride solution as represented by block 86. The magnesium chloride solution is then dried in a dryer as represented by block 88 and introduced into an electrolytic cell as represented by block 90 and thereafter converted to magnesium metal and chlorine gas. The mining of Magnesium metal from the raw saline seep water is economically justifiable since sea water which has been traditionally utilized to mine magnesium contains only approximately 1,300 milligrams per liter of magnesium while the brackish waters collected from the saline seeps have magnesium contents of from about 2,500 to 7,000 ml. per liter. The saline brines contain in addition to magnesium minable quantities of calcium sulfate or gypsum to provide byproducts that can be mined from the saline seep water before the water is purified and re-circulation back to the land or discharged into rivers and streams.

Detailed Description Text (18):

The pretreatment removal of the magnesium and calcium sulfate assists not only in providing more effective reverse osmosis but also allows less retention time in the reverse osmosis unit and less fouling. However, in the case of pre-treatment careful pH and sodium hydroxide control is necessary to insure the quality of the

purified magnesium hydroxide precipitate. The removal of the magnesium hydroxide precipitate assists in allowing the reverse osmosis unit to be smaller and run more economically and in an alternative embodiment allows the utilization of a smaller ion exchange unit which may be substituted for the reverse osmosis unit. An ion exchange system would utilize a two resin bed preferably employing a strong acid such as hydrochloric acid and a weak base combination such as ammonia. The causticization of ammonia chloride will occur with magnesium oxide followed by the hydrolysis of magnesium chloride in a recycle loop. Thermal energy could be utilized to recover ammonia and hydrochloric acid for reuse and the solid waste salts then recovered rather than a brine stream. Typical equations for an ion exchange flow system would be as follows:

Detailed Description Text (25):

A portion of the sample of the raw saline seep of Example 1 was analyzed for specific conductance and precipitated with sodium hydroxide. Results of the precipitation and percentage analysis of the major elements present in the raw saline seep precipitate as filtered are as follows:

Detailed Description Text (30):

In this Example a 50% concentrate of the brine solution of Example 3 was treated with sodium hydroxide to recover the major elements which were separated out of the raw saline seep water of Example 1 by reverse osmosis. The results of this treatment of concentrated brine obtained by reverse osmosis were as follows:

Detailed Description Text (32):

It has been found that by utilizing a stronger concentration of sodium hydroxide than was utilized in Example 2 and by allowing the sodium hydroxide more time to react with the magnesium produces a greater percentage of magnesium hydroxide precipitating from the raw saline seep of Example 1. In this Example, the treatment of the raw saline seep of Example 1 precipitated 21.43% of the magnesium as magnesium hydroxide.

CLAIMS:

2. The method for mining and reclaiming land of claim 1 wherein said step of purifying said subterranean water further includes the step of chemically treating said subterranean water with sodium hydroxide to precipitate said calcium and magnesium salts.
3. The method for mining and reclaiming land of claim 2 wherein said step of treating said subterranean water with said sodium hydroxide further includes a prior treatment of said subterranean water with a combination of sodium carbonate and sodium hydroxide to precipitate calcium as calcium carbonate before treating the filtrate with additional sodium hydroxide to remove magnesium salts as magnesium hydroxide.
4. The method of mining and reclaiming land of claim 3 wherein said step of purifying said subterranean water further includes the treatment of the resulting filtrate after the precipitation of said magnesium hydroxide with calcium hydroxide and sodium carbonate to remove excess calcium.
15. The process for simultaneously mining and reclaiming land of claim 11 wherein said step of mining comprises a step of collecting said waters in settling tanks and treating said collected waters with sodium hydroxide and sodium carbonate to first precipitate calcium carbonate.
16. The process for simultaneously mining and reclaiming land of claim 15 wherein said step of mining includes after said step of precipitation of calcium carbonate the removal of the mother liquid to a further settling tank that is treated with sodium hydroxide to precipitate magnesium hydroxide.

25. The method of mining salt laden glacial till while reclaiming a swale for agricultural purposes of claim 24 wherein said step of mining said collected water includes the step of pretreating said collected water with sodium hydroxide to form a precipitation of excess minerals.

26. The method of mining salt laden glacial till while reclaiming swale for agricultural purposes of claim 25 wherein said step of purifying said collected water includes the application of ion exchange to said collected water after said precipitation of excess minerals from said collected water after a pretreatment with sodium hydroxide.

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L2: Entry 37 of 51

File: USOC

Oct 31, 1967

DOCUMENT-IDENTIFIER: US 3350292 A
TITLE: Utilization of saline water

OCR Scanned Text (4):

United States Patent Office 31350,292 3,350,292 UTMIZATION OF SALIN'-P WATER Arthur Joseph Weinberger, Stamford, and Darwin Fiske De Lapp, New Canaan, Conn., assignors, by direct and mesne assignments, of one-half to American Cyanamid 5 Company, Stamford, Conn., a corporation of Maine, and one-half to the United States of America as repre. sented by the Secretary of the Interior Filed May 14, 1964, Ser. No. 367,428 11 Claims. (C]. 204-130) 10 ABSTRACT OF THF, DISCLOSURE Sea water is converted to potable form, and chemicals recovered, by adding a soluble alkali such as sodium hy- 15 droxide to convert bicarbonate to carbonate, and sodium , carbonate to precipitate calcium, the precipitates re- moved; magnesium separately separated by (1) cation exchange resi-@is, using recycled sodium chloride to regenerate, or (2) adding sodium hydroxide and separating 20 magnesium hydroxide; then acidifying, converting the de- scaled sea water to potable water and a saline concen- trate, and recovering salt or sodium hydroxide for re- cycle from the saline concentrate, and recovering bromine if desired. 25 This invention resulted from work done under a con- tract with the Office of Saline Water in the Department of the Interior entered into pursuant to the Saline Water Act, 30 42 U.S.C. 1951-1958g. This invention relates to processes for rendering saline wate rs potable and simultaneously recovering in com- merc ially usable form at least part of the mineral content of the water. Primarily, the invention is for the recovery 35 from sea water of potable wa'ter primarily for - drinking and domestic purposes, although also completelv - satisfac- tory for industrial and irrigation purposes. The - hardness, i.e., the Ca++ and Mg++, in the water is minimized to prev ent or reduce scale formation utilizing low cost 40 che micals, which preferably at least in part are regener- ated in the process to secure water sufficiently free from scale forming components that the water may be used in evap orators, and after concentration of the miicral con- tent, at least part of the mineral content is recovered, the 45 reco vered values being used in the process, or sold as such , or transformed to industrially saleable materials. In the past sea water has been distilled to yield potable wate r using a single effect or multiple effect evaporator or a multistage flash evaporator and at times generated 50 stea m has beei compressed to raise the iemperature so that the compressed steam could be used in part as the heati rig medium. Stich compressive distillation pro- cedures are particularly useful where power is available at a low figure. 55 In such distillation procedures the natural hardness of sea water has caused scaling of equipment, thus reducing capa city and increasing costs due to both the low heat trans fer coefficient aaid the cost of maintaining the equip- ment . 60 Also, in the past, efforts have been, made to recover com mercially useftil values from sea water. These have vari ed from schemes ranging from the exotic for recover- ing gold, to commercially useful processes for recover- ing magnesium, sodium chloride, or bromine. As illus- 65 trativ e of such prior art, U.S. Patent 2,764,472, W. R. Cady et al., "Brine Puri cation," Sept. , 1956, discloses a method for produch-ig substantially pure sodium chloride crystals. U.S. Patent 2,772,143, W. F. McIlbenny et al., "Method of Recovering Magnesium Salts From Sea 70 Water," Nov. 27, 1956, discloses a method for producing a magnesium salt from sea water in which the sea water Patented Oct. 31, 1967 2 is diluted with fresh water and the magnesium separated by an ion exchaige resin@ In this patent there is a disclosure of adding

sodium carbonate and sodium hydroxide to precipitate calcium carbonate and the effluent is fed to the ion exchange resin beds. U.S. Patent 2,934,419, V. Cook, "Method of Treating Sea Water," April 26, 1960, discloses the recovery of trace minerals from sea water by adding sodium hydroxide and sodium carbonate to precipitate calcium and magnesium, on the surface of which precipitate particles are concentrated trace elements. U.S. Patent 3,128,248, Suzuki, "Method For The Purification of Brine," April 7, 1964, shows the use of sodium hydroxide and lime or sodium carbonate to separate magnesium and calcium from brine and sea water, using as coagulant oil, soap or polyelectrolyte, with pressurizing of the system under air, thus forming, on release from pressure, fine air bubbles which give a more rapid flotation of the precipitated calcium carbonate and magnesium hydroxide. Since the times of the Phoenicians, or earlier, sea water has been evaporated to yield sodium chloride as table salt-sometimes of questionable purity. In each of these processes a single objective is controlled. We have now discovered a system whereby the sea water can be treated to yield potable water and mineral values concurrently; thus reducing the cost because part of the cost is borne by the value of the potable water and part of the cost is borne by the recovered chemicals. It has now been found that sea water can be descaled using chemicals obtained at least in part from the treated sea water so that a comparatively scale-free feed is obtained, thus permitting higher efficiency in the evaporator or other physical recovery process, and higher concentration of the effluent, which effluent is used as a source of chemicals. Both the chemicals and the potable water are regarded as saleable products; although the chemicals alone may be the primary product. The demand and transportation costs determine the economics in a particular location and determine which should be regarded as product and which should be regarded as by-product. The value of the chemicals produced and the cost of raw materials for the process varies with location. The material required in the largest quantity which is not yielded by the process is lime. The lime can, of course, be recovered by burning calcium carbonate, if a more economical source is not available. Similarly, even though the process is designed primarily using sodium hydroxide and sulfuric acid for pH control, and sodium chloride for ion resin exchange regeneration, there are times and places where the economics are warped by having the process streams from adjacent chemical plants which have useful constituents at distress prices. For example, if an acid such as hydrochloric acid or phosphoric acid, or a gaseous alkali such as ammonia or lithium hydroxide or potassium hydroxide is available where for peculiar local reasons the economic value is very low, such components can be introduced into the system. Lithium hydroxide or lithium carbonate or potassium hydroxide or potassium carbonate theoretically could be used but in any normal economic pattern are non-competitively priced. Similarly, ammonia could be used for pH control or introduced as ammonium carbonate with ammonia being recovered later in the process by treatment with lime. Such variations can be adjusted into the process with minor modifications in the presence of unusual economic considerations. The present process cannot be economically practiced on a small scale because the cost of labor would be proportionately too high. Where a major plant investment can be made to yield several million gallons of water per

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3 day, the process becomes economically more attractive. The process can become even more attractive where major quantities of heat are available from a process as, for example, a nuclear generating station or a large power plant, which makes heat available at a lower proportionate cost. Waste heat from plant processes is particularly attractive for distillation operations. Although the preliminary treatment can conveniently be at room temperature, it is frequently advantageous to warm the feed water to increase the speed of reaction, give a more readily filterable product, and reduce the viscosity during processing. Such heat is not wasted as the treated feed to later distillation would be of the water already heated, and the heat would be conserved. Such variations are, of course, within the scope of the present invention and in part depend on local economic factors, including climatic conditions, at any particular plant. As used in this

application, sea water refers essentially to the saline water that exists in the open sea, although it includes the same sea water, somewhat concentrated by partial evaporation in inland areas, such as the Red Sea, or as diluted by dumping minor quantities of chemicals or fresh water or sewage, as from rivers, to give a somewhat more dilute sea water, and sea water as diluted by melting ice as it exists in the polar regions. Although the chemical analysis of the sea water may vary somewhat in various areas of the world, the present process may be used successfully with any such sea water as feed. The feed is, of course, filtered or settled to remove seaweed or other solid particles, including fish, before use. Potable water is used to refer to water of a low solids concentration suitable for drinking or domestic purposes, such as the ordinary household uses, including washing the baby, washing clothes, dishes and other household chores. It is also satisfactory for the irrigation of crops, lawns, other agricultural uses and industrial uses, including boiler feed water, chemical processes, including plating baths, steel mills, cement mills, and ore processing. Usually the industrial and agricultural uses require a lower first cost so economics become important. The economics in each area must be considered as competitive sources and availability and purity affect the economic advisability of installing a plant for recovering potable water and chemical by-products from sea water. In some areas of the world, and even of the United States, the demand for potable water is already at the point that production from sea water is economically advantageous on a major scale. In other areas, potable water in streams and rivers is in such a large supply that sea water treatment is definitely out of the question for the near future. By physical conversion is meant the physical process of separating water from dissolved salts and includes the various forms of distillation, freezing, dialysis, reverse osmosis, and hydrate formation. In the drawings: FIGURE 1 is a flow sheet showing ion exchange separation of magnesium. FIGURE 2 is a flow sheet of an embodiment separating magnesium as the hydroxide, and salt recovered as such. @ FIG. 3 is a flow sheet showing magnesium separated as the hydroxide, and direct electrolytic regeneration of sodium hydroxide. In accordance with one modification of the present invention, saline water after preliminary screening to remove gross contaminants, is treated in any order with sodium hydroxide or lime and sodium carbonate to convert bicarbonate ions to carbonate ions and to precipitate calcium carbonate. The clarified solution is mixed with more sodium hydroxide to precipitate magnesium hydroxide which is thickened and either dried and calcined to yield magnesia as a product, or reacted with hydrochloric acid to convert it to magnesium chloride. The sea water, virtually free from calcium and magnesium, is fed to a distillation unit for the recovery of 3,350,292 potable water from the overhead and the concentrated saline effluent is further concentrated to yield crystalline sodium chloride, at least part of which is electrolyzed to produce sodium hydroxide and chlorine. As an alternate, the concentrated brine may be treated with lime to precipitate calcium sulfate, then sodium carbonate to precipitate excess calcium, then partly electrolyzed to yield anhydrous chlorine (collected as a product) and a sodium hydroxide solution containing some salt. A portion of this latter is treated with carbon dioxide gas (furnace stack gas flow in sulfur can be used) and recycled to the steps requiring sodium carbonate. Some of the sodium hydroxide is used as required in the treatment process, and any remainder sold. Part of the chlorine may be used to release bromine from the concentrated brine to obtain product bromine. Conveniently, alternatively, the sea water is treated with lime (calcium hydroxide) and soda ash (sodium carbonate) to precipitate out calcium carbonate. The calcium carbonate sludge can go to waste or be recovered if sufficient demand exists. After the carbonate is settled, the remaining solution is passed through a series of ion exchangers which pick up magnesium and any residual calcium ions. The softened water is fed to an evaporation unit. The effluent from the evaporation unit is evaporated further to obtain sodium chloride, preferably after sulfate removal. The ion exchange resin is regenerated with concentrated sodium chloride, which is made up by dissolving a portion of the crystallized sodium chloride produced in part of the fresh water. Concentrated brine from the sea water evaporation contains too much sulfate to be used for regeneration, since calcium sulfate would precipitate.

, It with the magnesium hydroxide when lime is added. A mixture of the brine with sodium chloride dissolved in fresh water may be used, so long as the sulfate present is not sufficient to precipitate calcium sulfate. The rich regenerant from the ion exchanger contains the major part of the magnesium and some calcium and excess sodium, as the chlorides. This is mixed with additional lime yielding magnesium hydroxide which is settled and/or filtered, dried and calcined to magnesium oxide or, where desired, converted to magnesium chloride with hydrochloric acid and electrolyzed for the recovery of magnesium metal. A very pure product is obtained, and can often be sold at a premium price. The overflow from magnesium hydroxide precipitation, containing substantial calcium and sodium chloride is mixed with the concentrate from the evaporator to yield calcium sulfate as a sludge and the supernatant solution is treated with sodium carbonate as required to separate excess calcium as calcium carbonate. If the maximum production of sodium chloride is not required, it is desirable to add only a portion of the overflow from magnesium hydroxide precipitation to the sea water concentrate, since the former contains more dissolved calcium than the equivalent of SO₄ in the latter. Other salts present in minor quantities can be separated out before or after the recovery of bromine if the economics at the location of operation justify recovery. Obviously, the exact proportions used will vary depending upon the economics of a particular plant. The costs of heat and the economic value placed on purified water and the market potential of various chemicals produced will vary depending on freight, purities, and other economic considerations. Accordingly, the examples below are to be considered as illustrative only and the exact operating points for maximum economic recovery varies from plant to plant and time to time but in accordance with the general principles outlined herein. It is to be understood that quantities of sea water treated are comparatively large and minor changes in economic conditions vary the preferred control points in the process. Example I As shown in FIGURE 1, sea water is treated with calcium oxide and sodium carbonate to precipitate calcium

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5 carbonate. The calcium oxide is added in sufficient quantity to convert substantially all of the bicarbonate in the sea water to carbonates and sufficient sodium carbonate is added to react with substantially all of the calcium then present. Only a small amount of the magnesium precipitates and the calcium carbonate which precipitates contains predominant portions of the heavy metal impurities such as aluminum and iron as their hydroxides. The calcium carbonate is settled or otherwise separated. The treated water is passed through several successive beds of ion exchange resins which absorb most of the magnesium and any residual calcium while releasing sodium to the solution. The water thus formed can be considered as descaled as it has a very small proportion of scaling-forming constituents present. Conveniently, and customarily, the pH is adjusted with a mineral acid. The acidification renders certain of the salts more soluble and permits deaeration and concentration. The descaled sea water is concentrated in an evaporator, specifically in this example a multiple effect evaporator, to yield product water which is substantially pure and a concentrate. Because the water has been descaled, it is convenient to concentrate about ten-fold until the sodium chloride in the water is near saturation, thus forming a concentrate. The ion exchange resin bed which is heavily loaded with magnesium and contains some calcium is regenerated by passing through the bed a concentrated solution, preferably saturated, or sodium chloride in fresh water. It is preferred to use a comparatively pure sodium chloride dissolved in pure water. If the brine concentrate itself were used the high concentration of sulfate ions would introduce complications. The regenerant solution contains the magnesium and calcium primarily as a chloride with some sodium chloride and, in accordance with conventional practice, lime may be added either as calcium oxide or as a slurry of calcium hydroxide, which precipitates magnesium as the hydroxide; which is thickened, filtered and washed. It may be dried and calcined to give comparatively pure magnesia, or may be treated with hydrochloric acid to yield magnesium chloride for electrolysis to produce metallic magnesium or other purposes. The solution

from which the magnesia has been precipitated, containing a mixture of sodium and calcium chlorides, is mixed with a concentrate from the water conversion unit, containing sulfates, precipitating calcium sulfate which may be heated and treated to form a desired grade of gypsum. The clarified liquid from the calcium sulfate precipitation is treated with sodium carbonate to precipitate residual calcium as the carbonate, which can be washed, dried and calcined for use in the magnesia precipitation, or elsewhere. The clarified low calcium brine is evaporated to yield pure water and comparatively pure sodium chloride. Preferably the sodium chloride is separated out before dryness is reached and before any other components precipitate, so that a filtrate is obtained containing bromides and most of the residual salts not earlier accounted for. Some of the sodium chloride and pure water obtained are used to prepare the brine for regenerating the ion exchange resin. The concentrated brine effluent from salt evaporation is acidified with sulfuric acid, ebulliated to release free bromine from the dissolved bromides, then blown with air to displace the bromine as vapor. The bromine vapor is absorbed in sodium carbonate solution which is acidified and steamed in accordance with conventional practices to yield product bromine. After the bromine separation the brine is conveniently neutralized with sodium carbonate and discarded, although it may be discarded in the acid form. As is obvious, certain modifications may be made, as for example, the lime may be added as a slurry of calcium hydroxide, or alkalinity can be introduced as sodium hydroxide which reduces the amount of calcium carbonate to be settled. The choice of the use of lime or sodium hydroxide depends upon the availability and availability of these respective materials. In the pH adjustment sulfuric acid is normally used as the cheapest of the mineral acids. However, if part of the product sodium chloride is electrolyzed to yield sodium hydroxide and chlorine, part of the chlorine may be converted to hydrochloric acid and the hydrochloric acid used for acidification. If other chemical operations are conducted adjacent, either sulfuric or hydrochloric acid in an adequately pure form may be present as a byproduct waste stream and, hence, chargeable at a very low cost. The required sodium carbonate may at least in part be produced by absorbing carbon dioxide from stack gases with caustic soda. The treatment such as the calcium carbonate settling and ion exchange steps may be at an elevated temperature as at higher temperature reactions occur more rapidly, and the viscosity of the solution is reduced so that it is more easily handled. At the same time as the effluent is to be treated in a physical conversion unit, if a physical conversion process is chosen which involves evaporation, the heat used to warm the sea water is recovered. Useful data on presoftening and ion exchange is given in U.S. Patent 2,772,143, supra. The precipitated calcium carbonate is in a comparatively pure form, which is easily handled and, as such, may be more valuable in this form than as the burned and recycled lime. Example 2 This process descales water at a high concentration ratio and offers considerable flexibility in by-products. Magnesia and chlorine are necessarily produced. Purified sodium chloride, sodium hydroxide, chlorine and bromine are produced in a desired amount up to the maximum values in the feed water. As shown in FIGURE 2, the sea water is treated with recycled sodium hydroxide solution containing some sodium chloride from an electrolysis cell and sodium carbonate containing some sodium chloride from absorbing carbon dioxide from stack gas in the same caustic salt solution. Settling may be aided by the addition of small quantities, of the order of 1 to 2 parts per million, of a high molecular weight polyacrylamide and the calcium carbonate settled out as a sludge. The decalcified sea water is then mixed with additional sodium hydroxide solution which precipitates the magnesium as a hydroxide. Additional polyacrylamide may be added to aid in the settling of the magnesium hydroxide. The magnesium hydroxide is washed, filtered, dried, and may be calcined to yield purified magnesium oxide or treated with hydrochloric acid to yield magnesium chloride. The clear effluent from the magnesium hydroxide precipitate is acidified with sulfuric acid, decanted, then fed to the physical conversion step. A compression distillation stage was used to give pure water as product, and a concentrate nearly saturated in sodium chloride. Whereas the concentrate could be treated as in Example 1, by choice it was fed into a salt evaporator with pure water being collected in the overhead and

evaporation being continued forming 60 pure product sodium chloride' until the incipient precipitation of other salts. At this point the residual brine was mixed with a mineral acid and ebullorine gas for the recovery of bromine as described in Example 1. After the stripping of the bromine the residual brine was discharged 65 back into the sea. Part of the product sodium chloride was mixed with fresh water and fed to electrolysis cells to generate saleable chlorine gas, and sodium hydroxide solution, containing some residual sodium chloride, which was recycled to the process. 70. The proportion which is electrolyzed depends on the demand for chlorine and caustic at the plant location. It is not necessary to remove sulfates from the concentrate to the salt evaporator unless a high salt product rate is desired. If high salt production is desired, it is convenient to remove the sulfates as described in Example 1.

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7 The waste brine from the bromine stripper is preferably- neutralized with sodium carbonate or lime to reduce damage to aquatic life from the effluent. The process of this example is particularly attractive because the output of chlorine from a ten million gallon per day conversion plant would amount to less than 3% of the commercially shipped chlorine in the United States and, hence, could be readily absorbed by the market. Example 3 As shown in FIGURE 3, the raw sea water is treated with sodium hydroxide containing sodium chloride and sodium carbonate containing- sodium chloride recycled from electrolysis cells, and stack gas absorption, Calcium carbonate is separated out, and magnesium hydroxide is precipitated by the addition of additional caustic. The descaled sea water is fed through a deaerator after being mixed with mineral acid and the descaled, acidified sea water is physically converted by a multiple-effect multiple-stage conversion distillation unit to yield pure water and a concentrated brine. Part of this brine is purged to bromine recovery as above described, which purge removes recycled products other than the desired products. The remainder of the concentrate has lime added thereto in sufficient quantity to separate the sulfates as calcium sulfate, which is recovered in the form of gypsum and then sodium carbonate is added in sufficient quantity to precipitate substantially all of the remaining calcium as calcium carbonate, which gives a concentrated sodium chloride solution which is fed to the electrolysis units. In this process the calcium carbonate can be burned to lime and the stack gases used as a source of carbon dioxide in the production of sodium carbonate. As is obvious to those skilled in the art, the processes described in these three examples may be in part interchanged with each other and the products can be separated out in the form which is acceptable to the market. For example, part of the sodium chloride can be sold as a brine where such brine is desired, and the cost of shipment is reasonable. The effluent from the bromine recovery contains the majority of trace minerals not otherwise accounted for. Suitable steps can be taken for their recovery if desired. Usually the material is not of sufficient value to warrant further treatment. Other changes and modifications obvious to those skilled in the art are within the scope of the appended claims. We claim: 1. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; subsequently separately separating magnesium ions from the decalcified sea water, thus forming a descaled sea water, adding a low cost mineral acid, separating the descaled sea water into a potable water product, and a saline concentrate, and recovering at least part of the sodium values in the sodium chloride in the saline concentrate in a form useful for recycling to the process, as at least one of (1) said soluble alkaline material and (2) said sodium carbonate. 2. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant

precipitated calcitirn carbonate, together with coprecipitated iron and aluminum as hydroxides; and subseqtiently separately adding additional sodium hydroxide and separating mag.nesium from the decalcified sea water, as the hydroxide, 3,350,292 13 thus forming a descaled sea water, addin.- a low cost mineral acid to the thus descaled sea water, evaporating and condensing part of the descaled sea water containing said mineral acid to form a potable water product and a residual saline concentrate, and recovering and recycling at least part of the sodium values in the saline concentrate as sodium hydroxide for pH adjustment. 3. The method of recovering potable water from sea water which comprises adding in any order to sea water 10 (1) a soluble alkaline material from the group consisting of sodi-Lim hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipi- 15 tated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; and subsequently separately separating magnesium ions from the decalcified sea water, with a cation exchange resin, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled 20 sea water, evaporating and condensing part of the descaled sea water containing said mineral acid to form a potable water product and a residual saline concentrate, and recovering and recycling at least part of the sodium values iri the saline concentrate as sodium chloride solution to 25 re- ,encrate said cation exchange resin. 4. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide 30 to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with- coprecipithted iron and aluminum as hydroxides; and subsequently separately 35 addin- additional sodium hydroxide and separating magnesium from the decalcified sea water, as the hydroxide, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically- converting the descaled sea water containing said mineral acid 40 to a potable water product and a saline concentrate b@ freezing, and recovering and recycling at least part of the sodium values in the saline concentrate as sodium hydroxide for pH adjustment. 5. The method of recovering potable water from sea 45 water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all 50 of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; and subsequently separately separating magnesium ions from the decalcified sea water, with a cation exchange resin, thus forming a descaled sea 55 water, adding a low cost mineral acid to the thus descaled sea water, pbsically converting the descaled sea water containing said mineral acid to a potable water product and a saline concentrate by freezing, and recovering and recycling at least part of the sodium values in the saline 60 concentrate as sodium chloride solution to regenerate said cation exchan,@e resin. 6. The method of recovering potable water frorn sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting 65 of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron 70 and aluminum as hydroxides, and subsequently separately adding additional sodium hydroxide and separating magnesium from the decalcified sea water, as the hydroxide, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically convert- 75 ing the descaled sea water containinf-, said mineral acid to

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3,350,292 9 a potable water product and a saline concentrate by eleetrodialysis, and recovering and reeycling at least part of the sodium values in the saline

concentrate as sodium hydroxide for pH adjustment. 7. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; and subsequently separately separating magnesium ions from the decalcified sea water, with a cation exchange resin, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically converting the descaled sea water containing said mineral acid to a potable water product and a saline concentrate by electrodialysis, and recovering and recycling at least part of the sodium values in the saline concentrate as sodium chloride solution to regenerate said cation exchange resin. 8. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides, and subsequently separately adding additional sodium hydroxide and separating magnesium from the decalcified sea water, as the hydroxide, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically converting the descaled sea water containing said mineral acid to a potable water product and a saline concentrate by reverse osmosis, and recovering and recycling at least part of the sodium values in the saline concentrate as sodium hydroxide for pH adjustment. 9. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; and subsequently separately separating magnesium ions from the decalcified sea water, with a cation exchange resin, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically converting the descaled sea water containing said mineral acid to a potable water product and a saline concentrate by reverse osmosis, and 10 recovering and recycling at least part of the sodium values in the saline concentrate as sodium chloride solution to regenerate said cation exchange resin. 10. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides, and subsequently separately adding additional sodium hydroxide and separating magnesium from the decalcified sea water, as the hydroxide, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically converting the descaled sea water containing said mineral acid to a potable water product and a saline concentrate by hydrate formation, and recovering and recycling at least 20 part of the sodium values in the saline concentrate as sodium hydroxide for pH adjustment. 11. The method of recovering potable water from sea water which comprises adding in any order to sea water (1) a soluble alkaline material from the group consisting of sodium hydroxide, calcium oxide and calcium hydroxide to convert substantially all of the bicarbonate to carbonate, and (2) sodium carbonate to react with substantially all of the calcium, and separating out the resultant precipitated calcium carbonate, together with coprecipitated iron and aluminum as hydroxides; and subsequently separately separating magnesium ions from the decalcified sea water, with a cation exchange resin, thus forming a descaled sea water, adding a low cost mineral acid to the thus descaled sea water, physically converting the descaled sea water containing said mineral acid to a

potable water product and a saline concentrate by hydrate formation, and recovering and recycling at least part of the sodium values in the saline concentrate as sodium chloride solution to regenerate said cation exchange resin. 40 References Cited UNITED STATES PATENTS 2,342,062 2/1944 Schenk ----- 202-205 45 2,934,419 4/1960 Cook ----- 71-1 3,017,338 1/1962 Butler et al ----- -- 204-128 3,055,734 9/1962 Pomykala ----- 23-201 3,075,828 1/1963 Kato et al - ----- 23-201 3,128,248 4/1964 Suzuki ----- 210-53 XR 50 3,239,460 3/1966 Popper ----- 210-24 JOSEPH SCOVRONEK, Primary Examiner. MORRIS O. WOLK, Examiner. E G V@HITBY, Assistant Examiner. 55

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File: USOC

Apr 16, 1946

DOCUMENT-IDENTIFIER: US 2398743 A

TITLE: Recovery of magnesium compounds

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Patented Apr. 16t IM 2939gt743 UNITED STATES PATENT OFFICE 2,398,743 RECOVERY OF MAGNESHM COMPOUNDS Sheldon B. Heath and Forest R Minger, Midhmd, Mich., assignors to TYAE D@@ Chemical Com- pany, Mdland, IMeh., a corporation of Bgch- Appucation Aprfl 24. 1944, Serial No. 532,388 7 Claims. (Cl. 23-91) This invention relates to a method of recover- ing magnesium chloride froni seb, water and other natural brineg containing the same. The principal object of the invention Is to pro- vide an improved method of treatin.g sea, water o and like brines@ to separate the magnesium salts dissolved therein and recover the same in the form of solid magnesium cliloride substantially free c)f the impurities normally present in such brines. Other obj@cts will be apparent from the 10 description. The process of the invention is based on a se-, ries of reactions which include raixing the sea water or other magnesium-containing brine with alkah, thereby precipitating insoluble magnesium 16 hydroxide, separating the latter from the spent brine, converting the separated hydroxide to mag- nesium chloride solution by neutralization with hydrochlori c acid, and then evaporating the solu- order tO Gbtaiiii a ftal pr6duct of high purity is hereinafter,@et forth In detail. The inv6tion may best be explained with reference to the accompanying drawing, which il- 25 lustrates diagramynatically the flow of materials in a preferred embodiment of the new process. The raw material, e. g. sea water or any of a number of natural inland brines, bitterns, and quantity of boron retained as impurity in the set_tled magnesium hydroxide precipitate. Thus it has been found that, when an amount of lime or other alkali is used which is less than. sufficient to react with all the magnesium salts in the sea water, the precipitated magnesium hydroidde carries with it, probably by adsorption, a large proportion of the boron compouyads in the rgw water.. - These compounds persist through the neutralizatioxx and subsequent steps of the process, and appear as impurities in the final magnesium chloride. However, if, for the precipitation of the magnesium hydroxide, an excess of alkali Is added, the boron compounds are not apPreciably retained by the magnesium hydroxide precipitate, but remain in solution and are dis. carded with the spent brine. Minimum retention of boron by the hydroxide results when the lime or other alkali is added in such excess as to pro. the like, invariably contains, in addition to the 39 hydroxide. The limit of solubility of calcium hydesired magnesium chloride, substantial prppor- droxide in sea water correspon to a above, preferably about 0.015 N to 0.03 N; wftth lime, these veaues correspond roughly to a 10 to 30 per cent overliming of the sea water. A higher alkalinity, e. g. up, to the limit of solubuity of calcium hydroxide in the brine, does not unfavorably affect the operation of the process, although it has no material advantage as regards the reduction of the boron content of the niaguestuni tion to form solid magnesium chloride. The man- 20 duce in the aqueous suspension a soluble alkalinner in which these steps are to be carried out in ity corresponding to a normality of 0.01 N or tions of dissolved calcium salts and of sodium chlorid6. In most cases small but signifleaut pro- portions of other substances, such, as sulfates, car- bonates, and borates, are also present. All these 35 materials are, of course, to be separated from the magnesium ebloride. In the process as shown in the drawing, the raw sea

water is run into a flocculator I in which it is mixed with an aqueous solution or suspension of an alkali conveniently milk of Lime which is prepared by slaking quicklime in water, classifying to remove sediment, and thickening the resulting mixture to a concentration of 10 to 15 pounds of lime per cubic foot. The alkaline converts the dissolved magnesium salts in the sea water to insoluble magnesium hydroxide, the latter forming as a flocculent precipitate which is separated from the sea water and converted to magnesium chloride in the subsequent stages of the process. In the case of sea water and other brines which have an appreciable content of dissolved boron compounds, careful control of the proportion of SIM employed is necessary to minimize the alkalinity of about 0.04 N, while with stronger brines the solubility of calcium hydroxide may rise to a soluble alkalinity of as much as 0.065 N. Following the precipitation step in the flocculator I the treated sea water is transferred to a settling tank 2, such as a Dorr thickener, in which settling is effected in a quiescent state, so that the magnesium hydroxide settles to the bottom of the tank as a thick sludge, which is withdrawn when it contains from about 5 to 15 per cent by weight of anhydrous magnesium hydroxide. The bulk of the sea water remains as a supernatant clarified liquor, which is ordinarily allowed to overflow to waste. The thickened magnesium hydroxide sludge from the settling tank 2 is pumped to a filter 3, conveniently a suction filter of the Moore type, in which the magnesium hydroxide is recovered as a cake, the filtrate being discarded or returned to the flocculator for reworking. The wet filter cake of magnesium hydroxide, containing small amounts of calcium salts, sodium chloride, soda, and other impurities, is forwarded for

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hydrochloric acid and the remainder with a hydrochloric-sulfuric acid mixture, and then recombining the two neutralized brines, it may be convenient to neutralize both portions of the creamed slurry with mixtures of hydrochloric and sulfuric acids one portion with a mixture containing a high proportion of hydrochloric acid and the other with an acid mixture containing mostly sulfuric acid, and then to combine the two neutralized liquors to effect calcium sulfate adjustment, as described. Again, in another modification, after the magnesium hydroxide slurry resulting from the creaming step has been divided into two portions, the one is neutralized with a hydrochloric acid-sulfuric acid mixture and the other with hydrochloric acid alone, the two resulting neutralized brines then being evaporated separately, after which the concentrated brines are mixed in measured proportions to remove calcium and sulfate. In further modifications, the creamed magnesium hydroxide slurry may be acidified in one step with a mixture of the two acids in controlled proportions or the entire mixture may be acidified with hydrochloric acid alone, and a soluble sulfate thereafter added to react with the dissolved calcium salts in the adjusting step, either prior to or subsequent to evaporation to 30 to 40 per cent magnesium chloride concentration. The removal of calcium salts and sulfate from the neutralized magnesium chloride brine may also be effected according to the invention by methods differing slightly in detail from the careful calcium-sulfate adjusting operation already described. For instance, the neutralization may be carried out with hydrochloric acid or hydrochloric and sulfuric acid mixtures in such manner as to produce a substantial equivalency of calcium and sulfate in the neutralized magnesium chloride brine, thus insuring removal of most of the calcium and sulfate as calcium sulfate precipitate, but leaving, however, a slight excess of calcium or sulfate, as may be convenient. Then the resulting brine, after filtration or settling to remove the calcium sulfate already precipitated, silt, etc., and preferably but not necessarily also after evaporation to 30-40 per cent magnesium chloride concentration, may be later treated with a strong calcium chloride solution or with a strong soluble sulfate solution, e. g. sodium sulfate, in a proportion sufficient to produce substantially exact equivalency of calcium and sulfate. The small additional amount of calcium sulfate so formed is then removed, leaving a practically pure magnesium chloride solution. In an alternative procedure, useful when a slight amount of excess sulfate is present in the evaporated neutralized brine, the bulk of precipitated calcium

sulfate is removed as explained, after which a solution of a barium salt, e. g. barium chloride, is added in a proportion just sufficient to remove the dissolved sulfate as barium sulfate precipitate. In the foregoing process description milk of lime, i. e. calcium hydroxide, has been referred to as the alkaline precipitant for magnesium hydroxide. Either high calcium lime or dolomite lime may be used for the purpose. In some cases dolomite lime may be preferred for the reason that its magnesia content is added to the output of magnesium hydroxide and, in turn, of magnesium chloride. An alkali metal hydroxide, e. g. sodium hydroxide, may also be used, if desired. This application is a continuation-in-part of our prior application Serial No. 405,190, filed August 2, 1941. We claim: 1. In a method of treating sea water to recover magnesium salts therefrom as substantially pure magnesium chloride, the steps which comprise: treating the sea water with milk of lime in a proportion sufficient to react with all the magnesium salts in the sea water and to produce in the treated water an alkalinity above 0.01 N but not materially exceeding 0.03 N, thereby forming a precipitate consisting essentially of magnesium hydroxide but also containing small proportions of calcium salts and sodium chloride as impurities; separating the magnesium hydroxide precipitate from the treated seawater; creaming the precipitate with sufficient magnesium chloride brine containing 8 to 15 per cent by weight of magnesium chloride to form a suspension of magnesium hydroxide; dividing the creamed suspension into two portions; neutralizing one portion with aqueous hydrochloric acid in a quantity sufficient to produce in the neutralized solution a pH value of about 5.5 to about 7.0, forming magnesium chloride solution, and neutralizing the other portion with an aqueous mixture of hydrochloric acid and sulfuric acid in a quantity sufficient to produce in the neutralized solution a pH value about 5.5 to about 7.6, forming a solution of magnesium chloride containing soluble sulfate, both such solutions also containing small proportions of calcium salts and sodium chloride as impurities; combining the two neutralized solutions in such proportions that the resulting solution contains sulfate in a quantity substantially equivalent stoichiometrically to that of the calcium salts dissolved therein, removing insoluble impurities from the combined solution and evaporating the clarified solution to a concentration between about 30 and about 40 per cent by weight of magnesium chloride, at which concentration the calcium sulfate and sodium chloride impurities are substantially insoluble therein; agitating the evaporated solution for a period sufficient to permit the calcium sulfate and sodium chloride impurities to precipitate substantially completely; removing the precipitated impurities by filtration; and evaporating the filtered solution to recover the magnesium chloride therefrom in solid form. 2. In a method of treating brines containing magnesium and calcium salts to recover the magnesium content thereof as substantially pure magnesium chloride, the steps which consist in mixing with the brine an excess of alkali over the chemical equivalent of the magnesium salts therein but not substantially more than is required to produce in the treated brine a soluble alkalinity of 0.065 N, thereby forming a precipitate of magnesium hydroxide, separating the precipitate, creaming the separated precipitate with magnesium chloride solution, to produce a uniform suspension, neutralizing the suspension with aqueous hydrochloric acid and producing in the solution a concentration of sulfate substantially equivalent stoichiometrically to that of residual calcium salt impurities therein, to form a solution of magnesium chloride containing calcium sulfate together with sodium chloride as impurities, evaporating the solution to a concentration at which the calcium sulfate and sodium chloride are substantially insoluble therein, separating the insoluble impurities, and further evaporating the purified solution to recover magnesium chloride in solid form. 3. In a method of treating sea water to recover substantially pure magnesium chloride therefrom,

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4 2,898,743 the steps which consist in mixing with the sea water an excess of milk of lime over the chemical equivalent of the magnesium salts therein but not substantially more than is required to produce in the treated seawater a soluble alkalinity of 0.04 N, thereby forming a precipitate of magnesium hydroxide,

separating the precipitate, creaming the separated precipitate with magnesium chloride solution to produce a uniform suspension, dividing the suspension into two portions, neutralizing one portion with aqueous hydrochloric acid to form a solution of magnesium chloride, neutralizing the other portion with aqueous hydrochloric acid and sulfuric acid to form a solution of magnesium chloride containing insoluble sulfate, both such solutions also containing a small proportion of calcium salts as impurity, combining the two neutralized solutions in such proportions that the resulting solution contains sulfate in a quantity substantially 20 equivalent stoichiometrically to that of the calcium salts dissolved therein, evaporating the combined solution to a concentration at which calcium sulfate is substantially insoluble therein, filtering the evaporated solution, to remove such calcium sulfate, and further evaporating the filtered solution to recover the magnesium chloride therefrom in solid form. 4. In a method of treating natural brines containing magnesium salts together with a small proportion of boron compounds to recover magnesium chloride therefrom with a low content of boron as impurity, the steps which consist in adding to the brine an alkali in excess of the chemical equivalent of the magnesium salts therein sufficient to produce in the treated brine a soluble alkalinity between 0.01 N and 0.065 N, whereby a precipitate of magnesium hydroxide is formed having a low boron content, separating the precipitate, dissolving the separated precipitate in aqueous hydrochloric acid to form a substantially neutral solution of magnesium chloride, and recovering magnesium chloride from the latter solution. 5. In a method of treating natural brines containing magnesium salts together with a small proportion of boron compounds to recover the magnesium content thereof in a form having a low content of boron as impurity, the steps which consist in adding milk of lime to the brine in excess of the chemical equivalent of the magnesium salts therein sufficient to produce in the treated brine a soluble alkalinity between 0.01 N and that which corresponds to the limit of solubility of calcium hydroxide therein, whereby a precipitate of magnesium hydroxide is formed having a low boron content, and separating the precipitate from the treated brine. 6. In a method of treating sea water to recover the magnesium content thereof in a form having a low content of boron as impurity, the steps which consist in adding milk of lime to the sea water in excess of the chemical equivalent of the magnesium salts therein sufficient to produce in the treated sea water a soluble alkalinity between 0.01 N and 0.04 N, whereby a precipitate of magnesium hydroxide is formed having a low boron content, and separating the precipitate from the treated sea water. 1. In a method of treating natural brines containing magnesium salts together with a small proportion of boron compounds to recover the magnesium content thereof in a form having a low content of boron as impurity, the steps which consist in adding to the brine an alkali in excess of the chemical equivalent of the magnesium salts therein sufficient to produce in the treated brine a soluble alkalinity between 0.01 N and 0.065 N, whereby magnesium hydroxide is precipitated having a low boron content, and separating the precipitate from the aqueous liquor. SHELDON B. I. ARATH. FOREST R. NCENGER.

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Term:	L1 and sodium hydroxide and precipitation same	▲
	calcium hydroxide	▼

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<u>L2</u>	L1 and sodium hydroxide and precipitation same calcium hydroxide	51	<u>L2</u>
<u>L1</u>	sea water or seawater	58585	<u>L1</u>

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☐ 1. Document ID: US 20060065600 A1

L6: Entry 1 of 10

File: PGPB

Mar 30, 2006

PGPUB-DOCUMENT-NUMBER: 20060065600

PGPUB-FILING-TYPE:

DOCUMENT-IDENTIFIER: US 20060065600 A1

TITLE: Processes for recovering oligomers of glycols and polymerization catalyst from waste streams

PUBLICATION-DATE: March 30, 2006

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Sunkara; Hari Babu	Hockessin	DE	US
Cavall; James Richard	Newark	DE	US
Goudie; William Wayne	Kennett Square	PA	US

US-CL-CURRENT: [210/651](#); [210/652](#), [568/617](#), [568/621](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	K00C	Draw D
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☐ 2. Document ID: US 20060016754 A1

L6: Entry 2 of 10

File: PGPB

Jan 26, 2006

PGPUB-DOCUMENT-NUMBER: 20060016754

PGPUB-FILING-TYPE:

DOCUMENT-IDENTIFIER: US 20060016754 A1

TITLE: Water-treating microbicide, water treatment method and water treatment apparatus

PUBLICATION-DATE: January 26, 2006

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Ito; Akihiko	Otsu-shi		JP
Oto; Katsufumi	Otsu-shi		JP
Sugita; Kazuya	Otsu-shi		JP
Fusaoka; Yoshinari	Otsu-shi		JP

US-CL-CURRENT: [210/652](#); [210/696](#), [210/701](#), [210/764](#), [210/806](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw D
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☐ 3. Document ID: US 20050098499 A1

L6: Entry 3 of 10

File: PGPB

May 12, 2005

PGPUB-DOCUMENT-NUMBER: 20050098499

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20050098499 A1

TITLE: PROCESS FOR PRE-TREATING AND DESALINATING SEA WATER

PUBLICATION-DATE: May 12, 2005

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Hussain, Mohammed Azam	Houston	TX	US

US-CL-CURRENT: [210/639](#); [210/651](#), [210/652](#), [210/702](#), [210/774](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw D
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☐ 4. Document ID: US 20040065617 A1

L6: Entry 4 of 10

File: PGPB .

Apr 8, 2004

PGPUB-DOCUMENT-NUMBER: 20040065617

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20040065617 A1

TITLE: Methods for reducing boron concentration in high salinity liquid

PUBLICATION-DATE: April 8, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Wilf, Mark	San Diego	CA	US
Bartels, Craig R.	San Diego	CA	US
Hirose, Masahiko	Ritto		JP

US-CL-CURRENT: [210/652](#); [210/639](#), [210/696](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw D
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☐ 5. Document ID: US 20030230531 A1

L6: Entry 5 of 10

File: PGPB

Dec 18, 2003

PGPUB-DOCUMENT-NUMBER: 20030230531
PGPUB-FILING-TYPE: new
DOCUMENT-IDENTIFIER: US 20030230531 A1

TITLE: Method for reducing boron concentration in high salinity liquid

PUBLICATION-DATE: December 18, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY
Wilf, Mark	San Diego	CA	US
Bartels, Craig R.	San Diego	CA	US
Hirose, Masahiko	Ritto		JP

US-CL-CURRENT: [210/639](#); [210/641](#), [210/652](#), [210/696](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMNC	Draw D
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☐ 6. Document ID: US 6537456 B2

L6: Entry 6 of 10

File: USPT

Mar 25, 2003

US-PAT-NO: 6537456
DOCUMENT-IDENTIFIER: US 6537456 B2

TITLE: Method and apparatus for high efficiency reverse osmosis operation

DATE-ISSUED: March 25, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mukhopadhyay; Debasish	Palo Alto	CA	94306	

US-CL-CURRENT: [210/652](#); [210/638](#), [210/639](#), [210/650](#), [210/651](#), [210/661](#), [210/900](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMNC	Draw D
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☐ 7. Document ID: US 6113797 A

L6: Entry 7 of 10

File: USPT

Sep 5, 2000

US-PAT-NO: 6113797
DOCUMENT-IDENTIFIER: US 6113797 A

TITLE: High water recovery membrane purification process

DATE-ISSUED: September 5, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Al-Samadi; Riad A.	Burlington			CA

US-CL-CURRENT: 210/652; 210/650, 210/651, 210/660, 210/702, 210/705, 210/712,
210/723, 210/724, 210/749

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 8. Document ID: US 5925255 A

L6: Entry 8 of 10

File: USPT

Jul 20, 1999

US-PAT-NO: 5925255

DOCUMENT-IDENTIFIER: US 5925255 A

TITLE: Method and apparatus for high efficiency reverse osmosis operation

DATE-ISSUED: July 20, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mukhopadhyay; Debasish	Palo Alto	CA	94306	

US-CL-CURRENT: 210/652; 210/638, 210/651, 210/661, 210/663

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 9. Document ID: US 5501798 A

L6: Entry 9 of 10

File: USPT

Mar 26, 1996

US-PAT-NO: 5501798

DOCUMENT-IDENTIFIER: US 5501798 A

TITLE: Microfiltration enhanced reverse osmosis for water treatment

DATE-ISSUED: March 26, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Al-Samadi; Riad A.	Burlington			CA
Benedek; Andrew	Toronto			CA

US-CL-CURRENT: 210/652; 210/638, 210/641, 210/650, 210/651, 210/705, 210/712,
210/714, 210/749

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 10. Document ID: US 4036749 A

L6: Entry 10 of 10

File: USPT

Jul 19, 1977

US-PAT-NO: 4036749

DOCUMENT-IDENTIFIER: US 4036749 A

TITLE: Purification of saline water

DATE-ISSUED: July 19, 1977

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Anderson; Donald R.	Los Alamitos	CA	90720	

US-CL-CURRENT: 210/638; 203/7, 204/542, 210/259, 210/652, 210/726, 62/532

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	K00C	Draw D
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